



## Short communication

# Metal supported tubular solid oxide fuel cells fabricated by suspension plasma spray and suspension high velocity oxy-fuel spray

Yeong Yoo <sup>a,\*</sup>, Youliang Wang <sup>b</sup>, Xiaohua Deng <sup>a</sup>, Devinder Singh <sup>a</sup>, Jean-Gabriel Legoux <sup>b</sup>

<sup>a</sup> ICPE, National Research Council Canada, Ottawa, ON, Canada

<sup>b</sup> IMI, National Research Council Canada, Boucherville, QC, Canada

## HIGHLIGHTS

- Applied unique suspension thermal spray for metal supported tubular SOFCs for the first time.
- Utilized commercially available 430 SS sintered porous tubular supports with 1/4" diameter.
- Deposited thin and crack-free SDC electrolyte on the anode by suspension HVOF spray developed.
- Demonstrated scalable fabrication technologies for potential commercialization.

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We dedicate this manuscript to co-author Youliang Wang, who passed away on March 25, 2012 in a car accident in California, USA. He was a devoted and loving husband, and father of one.

### Keywords:

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## ABSTRACT

Low temperature (LT) metal supported solid oxide fuel cells (SOFCs) have many advantages in comparison to conventional electrode or electrolyte supported type SOFCs. NRC has demonstrated high performance LT metal supported planar SOFCs fabricated by either wet colloidal spray/sintering or suspension thermal spray. The combination of tubular configuration and metal supported SOFCs may produce more unique and very attractive advantages such as easy and inexpensive sealing method and materials, high specific and volumetric power density, cost-effective fabrication, enhanced robustness, rapid start up, red-ox cycle tolerance and potential use for a pressurized integrated system.

In this paper, thin film solid electrolyte of  $\text{Sm}_{0.2}\text{Ce}_{0.8}\text{O}_{1.90}$  (SDC) and  $\text{NiO-SDC}$  composite anode on sintered porous tubular metal supports were deposited by suspension HVOF spray and suspension plasma spray, respectively on sintered porous tubular metal support.  $\text{La}_{0.6}\text{Sr}_{0.4}\text{Co}_{0.2}\text{Fe}_{0.8}\text{O}_{3-\delta}$  (LSCF) cathode on the SDC electrolyte was formed by wet colloidal spray and subsequent sintering process as the final fabrication step. The detailed investigation of suspension and process-related parameters for suspension thermal spray was performed in order to produce thin and crack-free SDC thin film coatings. The electrochemical performance of single cells was demonstrated.

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## 1. Introduction

Growing environmental pressures have led to a need to develop cleaner and more efficient energy generation systems such as fuel cells. Solid oxide fuel cells (SOFCs) have a variety of advantages such as high efficiency for cogeneration of heat and electricity, the utilization of hydrocarbon-containing fuels, and applicability to a wide range of applications from small portable scale to large sized power systems. SOFCs may achieve efficiencies of 60% in stand-

alone condition and over 80% (net) if waste heat is used for cogeneration.

The main obstacles to the commercialization of solid oxide fuel cells are high production costs resulting from the need to use expensive materials, the complexity of the peripheral systems, and the complexity of the manufacturing processes required for the fabrication of SOFCs. Low temperature (LT) metal supported SOFCs have many advantages such as low material cost, excellent mechanical stability, rapid start up, potential red-ox cycle tolerance and potentially the avoidance of glass ceramics as sealant in comparison to conventional electrode or electrolyte supported type SOFCs [1]. Steele has shown that single cells incorporating thin

\* Corresponding author. Tel.: +1 613 993 5331; fax: +1 613 991 2384.

E-mail address: [yeong.yoo@nrc-cnrc.gc.ca](mailto:yeong.yoo@nrc-cnrc.gc.ca) (Y. Yoo).

Gd-doped ceria electrolyte may produce highly encouraging power density of 400 mW cm<sup>-2</sup> at 500 °C on H<sub>2</sub>O/CH<sub>4</sub> (1:1) [2]. Ceres Power in UK has demonstrated planar type LT metal supported SOFCs fabricated based on thick film (10–30 µm) electrolytes of Gd-doped ceria and perforated Ti-Nb-stabilized 17% Cr ferritic stainless steel [1,3,4]. Maximum power densities of 310 mW cm<sup>-2</sup> and 240 mW cm<sup>-2</sup> were obtained at 600 and 550 °C under moist hydrogen/air. The electrolyte was deposited using an electro-photoretic deposition process and the electrodes were deposited by wet spraying or screen-printing. However, the fabrication of high performance and reliable planar metal supported SOFCs has been considered highly challenging nowadays even in industry. NRC-ICPET has also developed planar type LT metal supported SOFCs by using a variety of scalable and innovative processing technologies such as RF magnetron sputtering, pulse injection type wet colloidal spray developed at ICPET [5], laser drilling, and photo-electrochemical etching for fabricating cell components, resulting in demonstrating maximum power densities of 370 mW cm<sup>-2</sup> at 600 °C from single cells of Sm<sub>0.5</sub>Sr<sub>0.5</sub>O<sub>3-δ</sub> (SSC)//SDC/ScSZ//NiO-SDC//SS substrate under humid 50% H<sub>2</sub> in Ar (3% H<sub>2</sub>O) and air [6–10].

The above mentioned metal supported cells have been fabricated based on planar type configuration. However, the combination of tubular or flattened tubular type configuration and metal supported SOFCs may produce more unique and very attractive advantages such as reduced sealing problem including easy and inexpensive sealing method and materials, high specific and volumetric power density, highly cost-effective fabrication, enhanced robustness including tolerance for mechanical vibration, improved red-ox stability, rapid start up, and potential use for pressurized integrated systems including solid oxide electrolyser cells (SOECs).

Tubular form is a matured design for SOFCs which has several merit compared to planar cells. In the tubular concept, the sealing is confined to small areas at the ends of the tubes which are outside the active zone and at significantly lower temperature. This design offers simplicity in term of materials and processing for sealing. In addition, the seal area is negligibly influenced by any cyclic condition. The gas manifold can be straight forward in tubular stacks avoiding the need of high temperature seals. Using metal substrate as anode support and current collector can help reduce the potential drop at the anode side during current collection. Furthermore, owing to good thermal conductivity, ductility and toughness of metallic structure, metal supported cells can have better thermal shock and fatigue resistance compared to electrode or electrolyte supported cells.

In this paper, thin film solid electrolyte of Sm<sub>0.2</sub>Ce<sub>0.8</sub>O<sub>1.90</sub> (SDC) and NiO-SDC composite anode on sintered porous tubular metal supports were deposited by suspension high velocity oxy-fuel (HVOF) and suspension plasma spray, respectively on sintered porous tubular metal support. La<sub>0.6</sub>Sr<sub>0.4</sub>Co<sub>0.2</sub>Fe<sub>0.8</sub>O<sub>3-δ</sub> (LSCF) cathode on the SDC electrolyte was formed by wet colloidal spray and subsequent sintering process as the final fabrication step. The detailed investigation of suspension and process-related parameters for suspension thermal spray was performed in order to produce thin and crack-free SDC thin film coatings. The electrochemical performance of single cells was demonstrated.

## 2. Experimental

### 2.1. Sintered porous tubular metal support

Sintered porous stainless steel 430 metal tubes (Mott Corp., Farmington, CT, USA) were used as metallic supports for the SOFCs fabrication. The external length, diameter and thickness of the metal tube were 150 mm, 6.35 mm and 1.0 mm, respectively and its

porosity determined by the Archimedes method was 17.6%. It was demonstrated that sintered porous 430 stainless steel substrates were used for planar metal supported SOFCs with long term durability over 1000 h at NRC-ICPET [10]. The same stainless steel material was used for fabricating tubular metal supported SOFCs. The linear coefficient of thermal expansion (CTE) of sintered porous 430 tubes was 11.25×10<sup>-6</sup> K<sup>-1</sup> at the temperature between 25 °C and 600 °C, as determined by TA Instruments TMA 2940 in nitrogen with the heating and cooling rates of 5 °C min<sup>-1</sup>.

### 2.2. Fabrication of single cells by suspension thermal spray

The metal supported tubular single cells comprising of sintered porous 430 stainless steel support, Ni-SDC anode, SDC electrolyte, and LSCF cathode were fabricated by atmospheric suspension plasma spray and suspension HVOF spray for anode and electrolyte, respectively at NRC-IMI and wet colloidal spray for cathode at NRC-ICPET. NiO-SDC (50:50 wt%) composite anode layer with 40–60 µm thickness and 76 mm length on the porous metal tube was deposited by atmospheric suspension plasma spray with an axial feed injection torch (Axial III, Northwest Mettech). The suspension of the mixture of NiO (d<sub>50</sub> = 14.4 µm, 0.21 m<sup>2</sup> g<sup>-1</sup>, Inco) and SDC (d<sub>50</sub> = 1.2 µm, 8.9 m<sup>2</sup> g<sup>-1</sup>, Nextech Materials) powders was prepared in ethanol with ethylene glycol and polyethylenimine (MWt 25,000 Alfa Aesar) as additives. Surface temperature of the substrate, as monitored by a pyrometer, was controlled by rotating tubular substrates during the deposition. After the deposition of NiO-SDC anode, SDC electrolyte with 20–30 µm thickness and 100 mm length was deposited on the anode and metal to cover the whole anode surface and uncoated metal surface of each 12 mm length from both end of the anode by an HVOF DJ-2700 hybrid gun (Sulzer-Metco, Westbury, NY) with propylene fuel (C<sub>3</sub>H<sub>6</sub>) and a suspension comprising of SDC d<sub>50</sub> = 20 nm, 80–220 m<sup>2</sup> g<sup>-1</sup> (nGimat, Atlanta, GA), ethanol, ethylene glycol and polyethylenimine. The further details on the thermal spray and suspensions can be found elsewhere [11,12].

La<sub>0.6</sub>Sr<sub>0.4</sub>Co<sub>0.2</sub>Fe<sub>0.8</sub>O<sub>3-δ</sub> (LSCF) cathode with 20 µm thickness and 25–50 mm length was deposited on the SDC electrolyte by wet colloidal spray using a suspension comprising of LSCF (d<sub>50</sub> = 0.5 µm, 0.21 m<sup>2</sup> g<sup>-1</sup>, Praxair), iso-propanol, and ethyl cellulose. The suspension was prepared by high-energy ball milling (SPEX Cert-Prep, USA) for 1 h and the cathode deposited on the SDC electrolyte was sintered at 700 °C for 1 h in air for the electrochemical characterization of single cells.

### 2.3. Characterization

The electrochemical performance of single cells composed of LSCF//SDC//NiO-SDC//SS was evaluated in the temperature range of 500–650 °C under humid 50% H<sub>2</sub> in Ar (3% H<sub>2</sub>O) as the fuel gas and air as the oxidant gas. Impedance data were obtained at open circuit using an impedance/gain-phase analyzer Solartron SI 1260 and an electrochemical interface Solartron SI 1287 (Solartron Analytical, Farnborough, UK) over a frequency range 10<sup>-2</sup> to 10<sup>6</sup> Hz with an applied potential of 10 mV. The cross-sectional microstructures of single cells after the deposition of cell components or cell testing were examined by field emission scanning electron microscope (SEM, Hitachi S4700).

## 3. Results and discussion

### 3.1. Deposition of anode and electrolyte on porous tubular metal supports

NiO-SDC and SDC thin films were individually deposited on sintered porous tubular metal supports by suspension plasma spray

and suspension HVOF spray, respectively as shown in Fig. 1. The deposition of NiO-SDC and SDC on planar type metal supports by suspension thermal spray was already demonstrated at NRC [11,12], but the difference of support configuration, surface temperature distribution and control, and support cooling method for the deposition of anode and electrolyte on tubular type metal supports may cause difficulty in fabricating a highly dense and defect-free electrolyte layer on NiO-SDC anode deposited on a tubular metal support. In comparison to conventional thermal spray or HVOF spray, suspension plasma or HVOF spray has higher efficiency and deposition rate. In addition, the use of a liquid feed carrier that permits feeding and spraying of much finer particles results in forming thinner coatings with more refined microstructure and grain size [11], but the particle size distribution and stable dispersion of powders in the suspension during feeding as well as the optimization of spray process parameters to make sure sufficient melting, but no decomposition or vaporization of spraying powders are essential to deposit thin films with intended phases and mechanical integrity.

Fig. 1a shows the vertical defects formed in SDC electrolyte layer during HVOF process. By controlling surface temperature distribution, support cooling method, and process parameters for the suspension plasma spray and suspension HVOF spray deposition of anode and electrolyte for tubular metal supports, the half cells with much less defective NiO-SDC anode with around 60  $\mu\text{m}$  thickness and SDC electrolyte with 25  $\mu\text{m}$  thickness on a sintered

porous tubular metal support were successfully fabricated for further performance characterization as shown in Fig. 1b. It was also very effective to make smooth surface of metal support and anode by sandblasting and mechanical polishing, respectively for reducing or eliminating mechanical cracking in the ceramic thin layers.

The controlled parameters for depositing defect-free SDC on NiO-SDC anode are summarized in Table 1. At these conditions, an average particle temperature close to 2850  $^{\circ}\text{C}$  that is higher than the melting point of ceria,  $\sim 2480$   $^{\circ}\text{C}$  [14] was measured. Even if the use of a liquid feed carrier in suspension plasma spraying (SPS) permits feeding and spraying of fine particles to form thin coatings with refined microstructure and grain size, the small particles created in the SPS carry little momentum and low thermal inertia, therefore, requiring heating far above their melting point in the high temperature plasma ( $T_{\text{flame}} > 8000$   $^{\circ}\text{C}$ ) to retain the melting temperature and high velocity further downstream at the impact on the substrate to form a dense coating. However, this high temperature in the chemically reducing plasma can lead to the transformation of  $\text{CeO}_2$  to  $\text{Ce}_2\text{O}_3$  which has lower melting and boiling temperatures, resulting in ceria decomposition and vaporization that limit deposition efficiency, electrochemical performance, and mechanical integrity of the electrolyte [11]. These effects can be alleviated under the condition that the ceria particles are heated slightly above their melting point by employing a much lower flame temperature of a high velocity oxy-fuel (HVOF) system (2600–3200  $^{\circ}\text{C}$ ).

In order to deposit defect-free SDC on NiO-SDC anode supported by porous metallic tubes as shown in Fig. 1b, the surface temperature gradient and distribution of substrates during the deposition process is also important to prevent mechanical cracking in the anode and electrolyte, resulting from residual thermal stress formed by localized heating. The further details on the effect of temperatures of plasma flame and particles on defect formation can be found elsewhere [11]. The optimized rotation speeds of 1700 and 560 rpm for tubular substrates were applied to control the surface temperature of the substrates, as monitored by a pyrometer, to below 700  $^{\circ}\text{C}$ , resulting in the reduction of the localized thermal stress in NiO-SDC anode and SDC electrolyte during suspension plasma spray and suspension HVOF spray, respectively. The suspension feeding rates for the NiO-SDC anode and SDC electrolyte were 3 and 2  $\text{kg h}^{-1}$ , respectively as shown in Table 1. Any sensitivity analysis for depositing defect-free electrode and electrolyte by suspension plasma spray and suspension HVOF spray hasn't been conducted yet.

### 3.2. Cell performance of single cells

Fig. 2a shows a tubular single cell assembled for electrochemical characterization and two sintered porous metal supports with 6" length and  $\frac{1}{4}$ " diameter. A ceramic cap covered on one end of

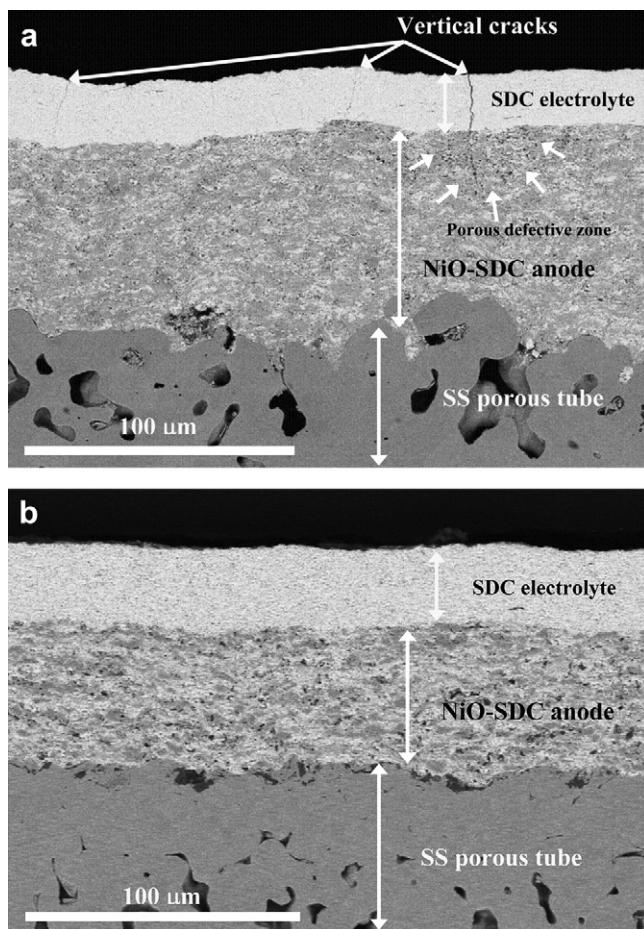


Fig. 1. Scanning electron micrograph of the fracture cross-section of tubular cells with (a) and without (b) vertical cracking in anode and SDC electrolyte fabricated by suspension plasma and suspension HVOF spray, respectively.

Table 1  
DJ-2700 HVOF spray parameters.

Parameter	Value
Propylene flow	85 slpm
Oxygen flow	279 slpm
Air flow	202 slpm
Carrier gas ( $\text{N}_2$ ) flow	20 slpm
Spray distance	15.2 cm
Suspension solid content	2.5 wt.%
Suspension feed rate	2 $\text{kg h}^{-1}$
Particle size	$\sim 20$ nm SDC
Suspension composition	70/30 wt.% glycol/ethanol

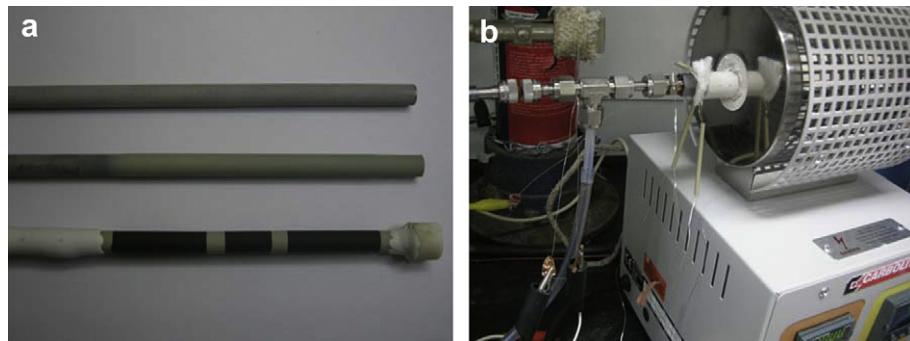


Fig. 2. Photos of a tubular single cell and two sintered porous metal supports with 6" length and 1/4" diameter (a) and single cell test in a horizontal tube furnace (b).

a tubular cell was fixed by alumina cement (Ceramabond<sup>TM</sup>503, Aremco Products Inc., USA) and a gas feeding fixture was directly connected to the single cell using Swagelok connector for single cell test. Fig. 2b shows a single cell with gas feeding components installed in a horizontal tube furnace for characterizing cell performance.

Fig. 3 shows the electrochemical performance of a single cell composed of LSCF//SDC//NiO-SDC//SS in the temperature range of 600–650 °C under humid 50% H<sub>2</sub> in Ar (3% H<sub>2</sub>O) as the fuel gas and air as the oxidant gas at a flow rate of 80 mL min<sup>-1</sup>. The open circuit voltages were around 0.88 V at 600 °C indicating negligible gas leakage through the electrolyte. A power density of 165 mW cm<sup>-2</sup> at 0.6 V was obtained at 600 °C under 50% H<sub>2</sub> in Ar (3% H<sub>2</sub>O). If each single cell has LSCF cathode with 75 mm length on a tubular support with 1/4" diameter, approximately 40 or 200 tubular cells would be required to build a 100 W portable or 500 W small scale stationary SOFC system, respectively. The output voltage can be adjusted to modify the connection of sub-modules both in series and in parallel for portable or stationary applications. In case of planar type metal supported SOFC with SDC electrolyte and 5 cm × 5 cm dimensions fabricated by the same suspension HVOF spray deposition, a power density of 230 mW cm<sup>-2</sup> at 0.6 V was obtained at 600 °C under 100% H<sub>2</sub> (3% H<sub>2</sub>O) [12]. In addition, the modeling data for Gd-doped ceria electrolyte based planar type metal supported single cells indicated that the power density of 200 mW cm<sup>-2</sup> can be achieved at 570 °C under 50% H<sub>2</sub> in Ar (3% H<sub>2</sub>O) [4]. Tubular type cells typically exhibit lower areal and volumetric power densities compared to planar type cells [13], therefore, it was confirmed

that the cell performance of metal supported tubular cells was reasonable.

Fig. 4 shows the impedance response for the single cell under open circuit voltage at temperatures of 600, 625 and 650 °C. The impedance spectra contain multiple depressed semicircles that are typical for single cell-related impedance spectra. It was determined that the ohmic resistance and electrode polarization resistance of the single cell were approximately 0.9 Ohm cm<sup>2</sup> and 0.5 Ohm cm<sup>2</sup>, respectively at 600 °C. In comparison to the ohmic resistance and polarization resistance of planar type metal supported cells fabricated at NRC [6,9], both of the ohmic resistance and electrode polarization resistance of tubular single cells were higher than those of planar single cells, indicating that the thicknesses of SDC electrolyte, NiO-SDC anode, and oxide scale on sintered porous metal substrate should be decreased and the microstructure of metal substrates and electrodes as well as interfacial contact should be optimized for fabricating high performance single cells. The ohmic polarization and activation polarization are temperature dependent mainly by virtue of the thermally activated dependence of SDC ionic resistivity and exchange current density, respectively. The concentration polarization, however, is weakly dependent on temperature [15]. As shown in Fig. 4, the low frequency arc has very weak temperature dependency in the temperature range of 600–650 °C and results from the limited mass transfer of air and fuel through electrode and sintered porous metal support.

Fig. 5 shows the degradation rate of the single cell at 600 °C and 150 mA cm<sup>-2</sup> under humid 50% H<sub>2</sub> in Ar (3% H<sub>2</sub>O) as the fuel

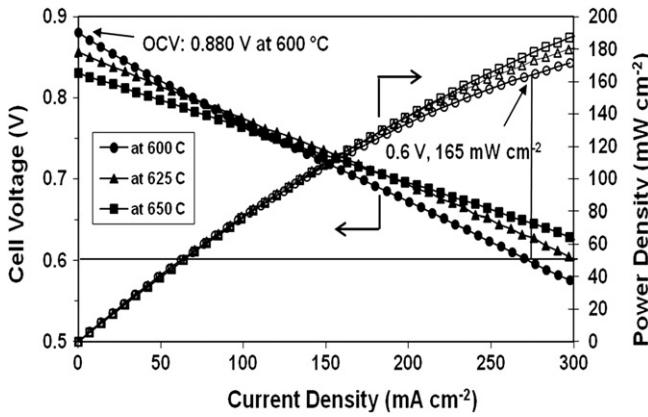


Fig. 3. I-V characteristics of  $\text{La}_{0.6}\text{Sr}_{0.4}\text{Co}_{0.2}\text{Fe}_{0.8}\text{O}_{3-\delta}$  (LSCF)//SDC//NiO-SDC at 600–650 °C under humid 50% H<sub>2</sub> in Ar (3% H<sub>2</sub>O) and air.

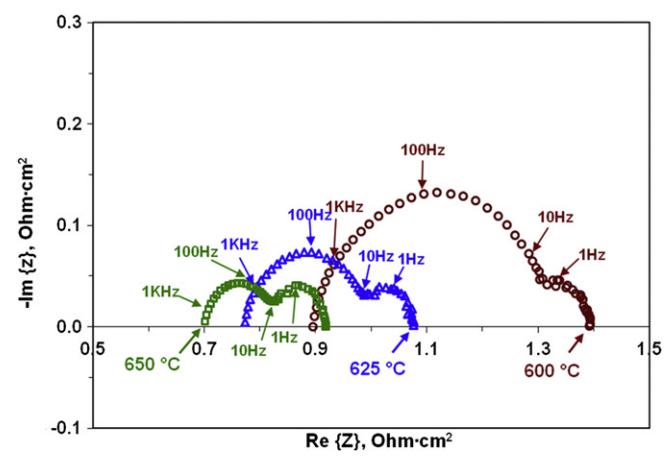


Fig. 4. Impedance spectra of LSCF//SDC//NiO-SDC//SS under open circuit voltage at 600, 625, and 650 °C.

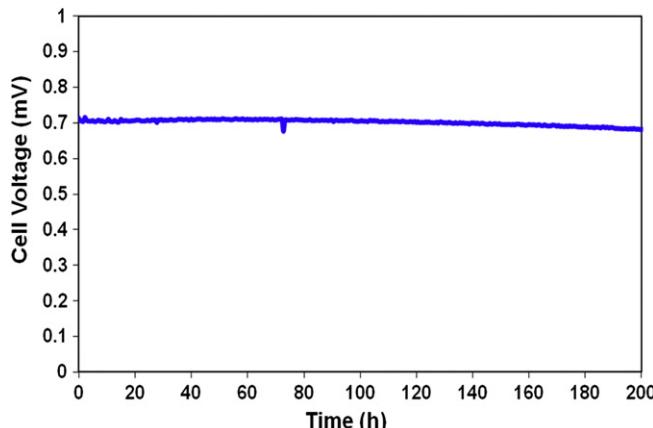


Fig. 5. Degradation of a metal supported tubular single cell at 600 °C and 150 mA cm<sup>-2</sup> under humid 50% H<sub>2</sub> in Ar (3% H<sub>2</sub>O) and air.

gas and air as the oxidant gas at a flow rate of 80 mL min<sup>-1</sup>. The cell performance was stable during the period of the durability test and the degradation rate of the single cell was less than 2% per 100 h during 200 h. Fig. 6a shows the electrochemical performance of the single cell before and after the degradation test at 600 °C during 200 h and Fig. 6b shows the impedance response for the single cell under open circuit voltage before and after the degradation test.

In order to improve the durability further, the detailed investigation of the high temperature oxidation behavior, pre-treatment and protective coating of porous metal substrates and the enhancement of quality of dense and defect-free electrolyte on the

whole metal tube should be considered. To utilize hydrocarbons as fuel and enhance fuel utilization for this tubular cell, it would be also interesting to consider the deposition of bilayer electrolytes such as Sc-doped zirconia (ScSZ)/SDC or Y-doped zirconia (YSZ)/SDC. The fabrication and characterization of bilayer electrolyte based metal supported tubular SOFCs have been in progress at NRC and the detailed results will be presented in the other paper to be published in this journal.

#### 4. Conclusions

Thin film solid electrolyte of Sm<sub>0.2</sub>Ce<sub>0.8</sub>O<sub>1.90</sub> (SDC) and NiO-SDC composite anode on sintered porous 430 stainless steel tubular metal supports with 6" length and 1/4" diameter were deposited by suspension HVOF spray and suspension plasma spray, respectively on sintered porous tubular metal support. La<sub>0.6</sub>Sr<sub>0.4</sub>Co<sub>0.2</sub>Fe<sub>0.8</sub>O<sub>3-δ</sub> (LSCF) cathode on the SDC electrolyte was formed by wet colloidal spray and subsequent sintering process as the final fabrication step. The maximum power density of 165 mW cm<sup>-2</sup> at 0.6 V and open circuit voltages around 0.88 V were obtained at 600 °C under 50% H<sub>2</sub> in Ar (3% H<sub>2</sub>O) as the fuel gas and air as the oxidant gas at a flow rate of 80 mL min<sup>-1</sup>. By controlling surface temperature distribution, support cooling method, and process parameters for the suspension plasma spray and suspension HVOF spray deposition of anode and electrolyte for tubular metal supports as well as lowering surface roughness of tubular metal support and anode for depositing electrolyte, the half cells with much less defective NiO-SDC anode with around 60 μm thickness and SDC electrolyte with 25 μm thickness on a sintered porous tubular metal support were successfully fabricated. The degradation rate of the single cell was less than 2% per 100 h during 200 h.

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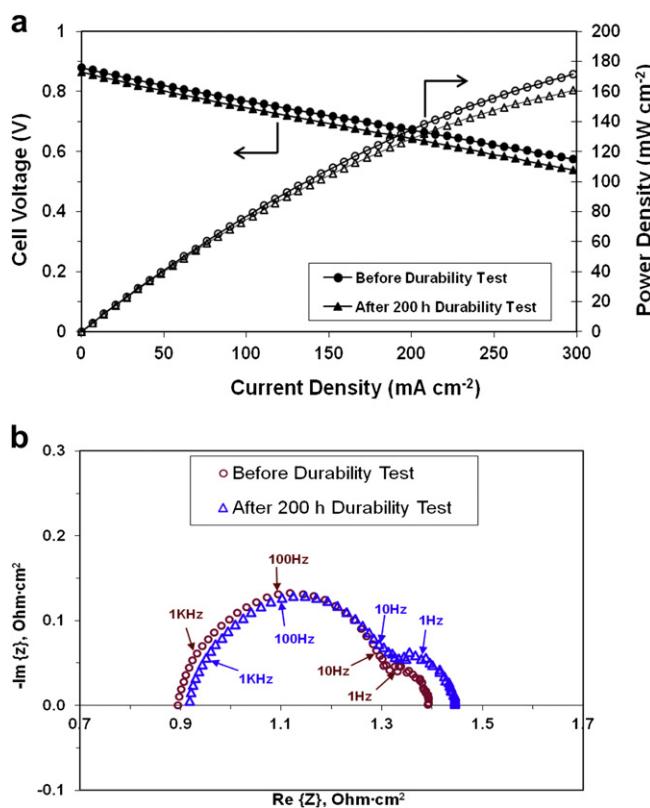


Fig. 6. I-V characteristics (a) and impedance spectra (b) of La<sub>0.6</sub>Sr<sub>0.4</sub>Co<sub>0.2</sub>Fe<sub>0.8</sub>O<sub>3-δ</sub> (LSCF)/SDC//NiO-SDC at 600 °C before and after 200 h degradation test.

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